JET PROPULSION LABORATORY
CALIFORNIA INSTITUTE OF TECHNOLOGY
PASADENA, CALIFORNIA

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MATERIAL SPECIFICATION
TWO PART STRUCTURAL EPOXY ADHESIVE
ELEVATED TEMPERATURE, AROMATIC AMINE CURED
(DIGLYCIDYL ETHER OF BISPHENOL A TYPE)

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N/C		2/9/66	

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1. SCOPE

1.1 This specification covers the qualification and acceptance requirements for the production, procurement, and testing of a spacecraft quality Two Part Structural Epoxy (Diglycidyl Ether of Bisphenol A Type) Adhesive, which cures at an elevated temperature with an aromatic amine curing agent.

2. APPLICABLE DOCUMENTS

2.1 The following documents, of the issue in effect on the date of invitation for bids, form a part of this specification to the extent specified herein:

SPECIFICATIONS

Jet Propulsion Laboratory

GMO-50234-PRS	Process Specification, Preparation of Surfaces for Adhesive Bonding
ZFT-4010-0007	Test Specification, Vacuum Weight Loss of Polymeric Materials

STANDARDS

American Society for Testing Materials (ASTM)

ASTM D 618-61	Conditioning Plastics and Electrical Insulating Materials for Testing
ASTM D 1002-64	Strength Properties of Adhesives in Shear by Tension Loading (Metal to Metal)
ASTM D 1084-55T	Consistency of Adhesives
ASTM D 1259-61	Nonvolatile Content of Resin Solutions
ASTM D 1652-62T	Epoxy Content of Epoxy Resins
ASTM D 1726-62T	Hydrolyzable Chlorine Content of Liquid Epoxy Resins
ASTM D 1780-62	Conducting Creep Tests of Metal to Metal Adhesives
ASTM D 1875-61T	Density of Adhesives in Fluid Form
ASTM D 2073-62T	Total, Primary, Secondary, and Tertiary Amine Values of Fatty Amines by Referee Potentiometric Method

3. REQUIREMENTS

3.1 Conflicting requirements. In case of conflict between the requirements of this specification and any document referenced herein, the requirements of this specification shall govern.

3.2 Qualification. A product furnished on contract or on order in accordance with this specification shall be a product which has been tested and which has passed the qualification tests specified in this specification. This product shall be listed as a qualified product herein.

3.3 Formulation changes. An adhesive shall be approved only for the formulation on which qualification tests are made. Any changes by the manufacturer, such as the adding of pigments, hardeners, carriers, dyes, fillers, extenders, plasticizers, oils, diluents, solvents, or other material; changing the type or form of the adhesive; changing the method of manufacture; or changing the mixing, application, or curing procedures shall require designating the adhesive as a new product which shall not be considered qualified and which shall require specific and separate qualification testing. The changed adhesive shall be given a new code number and shall meet all qualification requirements prior to use.

3.4 Identification of product. The manufacturer shall designate the adhesive base and curing agent by JPL Specification number and batch number. Trade names and/or code numbers may also be used to identify the materials.

3.5 Working characteristics. The following working characteristics shall apply.

3.5.1 Application. The mixed adhesive shall be capable of being readily applied at 25 ±5°C (77 ±9°F) and 50 ±25 percent relative humidity.

3.5.2 Pot life. The pot life of mixed adhesive shall be a minimum of four hours at 25 ±5°C (77 ±9°F).

3.5.3 Curing time and temperature. The material shall meet the requirements for the cured adhesive when cured for a maximum of 120 minutes at a maximum temperature of 177 ±3°C (350 ±5°F). The curing time and temperature shall be specified by the manufacturer in the instruction sheet. A step cure may be specified provided the temperatures and total cure time do not exceed the above requirements.

3.5.4 Curing pressure. Pressure during curing shall not be necessary to meet the requirements for cured adhesive. The manufacturer may recommend low pressures in the instruction sheet to facilitate mating of bonding surfaces, air squeeze-out, excess adhesive removal, and holding of adherends together during curing.

3.5.5 Shelf life. The unmixed adhesive base and curing agent shall be capable of meeting the requirements of this specification when stored for a minimum of 6 months after receipt at a minimum temperature of 25°C (77°F).

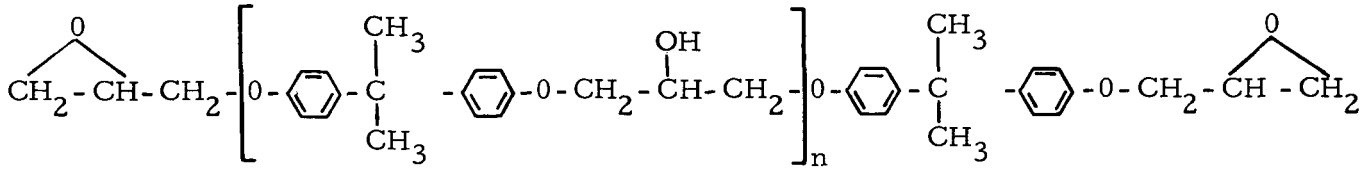
3.6 Instruction sheet. The manufacturer shall provide a dated, coded and titled instruction sheet, outlining instructions for the use of the adhesive, with the test report supplied when requesting qualification and also at least one copy for each container in each shipment of adhesive. The instruction sheet shall give the procedures in sufficient detail to meet all of the requirements of this specification.

3.7 Chemical characteristics. The chemical characteristics of the adhesive base and curing agent shall be as listed in Table I.

Table I. Chemical Characteristics

Material	No.	Property	Requirement	Test Procedure
Adhesive Base	1	Resin structure	Mfg. from Diglycidyl ether of Bisphenol A*	
	2	Epoxy Equivalent	310 - 360	Para. 4.6.1
	3	Hydrolyzable Halide Content	0.1% (maximum)	Para. 4.6.2
	4	Filler Type	Inorganic (metallic and/or ceramic)	
	5	Filler Content	40 - 47%	Para. 4.6.3
	6	Nonvolatile Content	99.5% (minimum)	Table IV
Curing Agent	7	Chemical species	Liquid aromatic amine or a liquid combination of aromatic amines	
	8	Amine Nitrogen	19 - 20%	Para. 4.6.4
	9	Hydrolyzable Halide Content	0.02% (maximum)	Para. 4.6.2
	10	Nonvolatile Content	98.5% (minimum)	Table IV

* The adhesive base resin shall have the following general polymer structure:



3.8 Physical properties. The physical properties of the adhesive base, curing agent, and cured adhesive shall be as listed in Table II.

Table II. Physical Properties

Material	No.	Property	Requirement	Test Procedure
Adhesive base	1	Viscosity	3000-6500 poise	Para. 4.6.5
	2	Density	1.47-1.57 g per milliliter	Table IV
	3	Infrared Spectrum	Closely similar to Fig. 1 *	Para. 4.6.6
Curing agent	4	Viscosity	4-7 poise	Para. 4.6.5
	5	Density	1.08-1.14 g per milliliter	Table IV
Cured adhesive	6	Vacuum weight loss		Para. 4.6.7
		After 100 ±4 hours exposure	1.00 percent maximum	
		After 200 ±8 hours exposure	1.05 percent maximum	

*Minor differences shall be acceptable provided no spurious absorption peaks in excess of 0.05 absorbance are present (see 4.6.6).

3.9 Mechanical properties. Aluminum specimens bonded with the adhesive shall have the mechanical properties listed in Table III.

3.10 Solvents and diluents. Solvents and/or diluents, whether reactive or nonreactive, shall not be combined with the adhesive materials.

Table III. Mechanical Properties of Bonded Aluminum Joints

No.	Property	Environmental Exposure	Test Temperature	Required Value	Test Procedure
1	Tensile Shear		24 ±3°C (75 ±5°F)	2900 psi average (minimum)	Table IV
2	Tensile Shear		149 ±3°C (300 ±5°F)	2000 psi average (minimum)	Table IV
3	Tensile Shear	192 hours at 149 ±3°C (300 ±5°F)	149 ±3°C (300 ±5°F)	2000 psi average (minimum)	Table IV
4	Tensile Shear		-55 ±3°C (-67 ±5°F)	2250 psi average (minimum)	Table IV
5	Tensile Shear	30 days at 49 ±3°C (120 ±5°F) and 95 ±5% relative humidity	24 ±3°C (75 ±5°F)	2100 psi average (minimum)	Table IV
6	Creep Rupture (1600 psi for 192 hours)		24 ±3°C (75 ±5°F)	0.015 inch deformation (maximum)	Table IV
7	Creep Rupture (800 psi for 192 hours)		149 ±3°C (300 ±5°F)	0.015 inch deformation (maximum)	Table IV

Table IV. Qualification Test Procedures

Test	Requirement Reference	Test Procedure
Epoxy Equivalent	No. 2 of Table I	Paragraph 4. 6. 1
Hydrolyzable Halide Content	Nos. 3 and 9 of Table I	Paragraph 4. 6. 2
Filler Content	No. 5 of Table I	Paragraph 4. 6. 3
Nonvolatile Content	No. 6 of Table I	ASTM D 1259-61 Method B (Solutions that release solvent slowly)
	No. 10 of Table I	
Amine Nitrogen	No. 8 of Table I	Paragraph 4. 6. 4
Viscosity	Nos. 1 and 4 of Table II	Paragraph 4. 6. 5
Density	Nos. 2 and 5 of Table II	ASTM D 1875-61T
Infrared Spectrum	No. 3 of Table II	Paragraph 4. 6. 6
Vacuum Weight Loss	No. 6 of Table II	Paragraph 4. 6. 7
Tensile Shear	Nos. 1, 2, 3, 4, and 5 of Table III	ASTM D 1002-64
Creep Rupture	Nos. 6 and 7 of Table III	ASTM D 1780-62
Shelf Life	Paragraph 3. 5. 5	Paragraph 4. 6. 8

3.11 Workmanship, purity, and consistency. The adhesive base, curing agent, and mixed adhesive shall have the following characteristics:

a. Shall be prepared in accordance with the best commercial practices.
b. Shall be essentially free of visible foreign matter and lumps.
c. Shall mix readily to a smooth paste.
d. Shall not separate or settle out over a period of eight hours.

4. QUALITY ASSURANCE PROVISIONS

4.1 Inspection responsibility. Unless otherwise specified in the contract or purchase order, the supplier is responsible for meeting all the requirements specified herein. JPL reserves the right to perform any of the inspections or tests set forth in this specification where such inspections or tests are deemed necessary to assure that supplies and services conform to prescribed requirements.

4.2 Classification of tests. The inspection and testing of the adhesives shall be classified as follows:

a. Qualification tests. Those tests that confirm the material as a qualified product.

b. Acceptance tests. Those tests that assure that each batch or shipment of material is essentially identical to material previously qualified.

4.3 Qualification tests.

4.3.1 Prior qualification. Unless otherwise specified by the JPL Materials Section, adhesives which have not passed the qualification tests, or adhesives which have passed the qualification tests and have been modified in any manner, shall satisfactorily pass the qualification tests prior to acceptance of any adhesive. All or any of the qualification tests may be repeated on material previously found satisfactory at any time at the option of the JPL Materials or Quality Assurance Sections. Failure to pass one or more of the tests shall be cause for rejection.

4.3.2 Qualification report. Unless otherwise specified by the JPL Materials Section, the manufacturer shall submit a dated and numbered report* proving the qualification of the adhesive to all of the requirements of this specification. Information concerning the products, processing of test specimens, test environments, test methods, test equipment, and test data shall be reported. In addition, either a statement indicating that the products have not been

*Consistent with the information supplied in 3.6.

rebranded shall accompany the samples, or if rebranded, the name of the original manufacturer and original designations of the products shall be indicated.

4.3.3 Tests. The qualification tests and the applicable test procedures shall be as listed in Table IV.

4.4 Acceptance tests. Unless otherwise specified by the JPL Materials Section, samples of each submitted batch of adhesive shall be subjected to and pass the acceptance tests listed in Table V.

Table V. Acceptance Tests

Test	Requirement Reference	Test Procedure
Viscosity	Nos. 1 and 4 of Table II	Paragraph 4.6.5
Density	Nos. 2 and 5 of Table II	ASTM D 1875-61T
Infrared Spectrum	No. 3 of Table II	Paragraph 4.6.6
Vacuum Weight Loss	No. 6 of Table II	Paragraph 4.6.7
Tensile Shear	Nos. 1 and 2 of Table III	ASTM D 1002-64

4.5 Test conditions for cured adhesive. All cured adhesive specimens shall be conditioned for a minimum of 24 hours, before exposure to specified testing or environmental exposure — test procedures, in a standard atmosphere having a temperature of 23 ±2°C (73 ±4°F) and a relative humidity of 50 ±5 percent per ASTM D 618-61. All tests or environmental exposure - test procedures, as applicable, shall be started within one hour after removing from the standard atmosphere. Tensile shear tests at 149 ±3°C (300 ±5°F) shall be conducted 10 minutes after the specimens have reached equilibrium with equilibrium being

reached in 3 to 10 minutes after starting heating. Specimen temperature shall not exceed the test temperature tolerance during the heat up period. Tensile shear tests at -55 ±3°C (-67 ±5°F) shall be conducted 10 minutes after the specimens have reached equilibrium.

4.6 Test procedures.

4.6.1 Epoxy equivalent. Epoxy equivalent shall be determined per ASTM D 1652-62T except the sample shall be prepared for titration as follows:

a. Proceed as described in paragraph 4.6.3 a., b., c., d. of this specification.
b. Close the side arm of the vacuum flask with a rubber dropper bulb. Insert a stirring bar, add 15 drops of crystal violet indicator solution, and wash down the inside wall with several milliliters of chlorobenzene.
c. Proceed with the titration per ASTM D 1652-62T.

4.6.2 Hydrolyzable halide content. Hydrolyzable halide content shall be determined as follows:

a. Prepare and hydrolyze a sample per ASTM D 1726-62T.
b. Add 10 milliliters of 1:1 nitric acid to the hydrolyzate and dilute with approximately an equal volume of distilled water.
c. Titrate with 0.1 N silver nitrate solution using a silver - mercurous sulfate reference electrode system as indicator.
d. Plot incremental milliliters of silver nitrate versus the emf of the electrode system to obtain the equivalence point.
e. Make a blank determination on the reagents following the same procedure but omitting the sample.

f. Calculate the halide content assuming all halide to be chloride as follows:

$$\% \text{ halide} = \frac{3.55N(A-B)}{W}$$

A = milliliters of silver nitrate solution required to titrate the sample.

B = milliliters of silver nitrate solution required to titrate the blank.

N = normality of the silver nitrate solution.

W = grams of sample used.

4.6.3 Filler content. The filler content of the adhesive base shall be determined as follows:

a. Weigh 10 - 20 gram (to the nearest 0.1 milligram) of adhesive base into a dry 500 milliliter beaker. Weigh 4 - 5 gram (to the nearest 0.1 milligram) of celite filter aid into the beaker. Add 125 - 150 milliliters of chlorobenzene into the beaker. Cover the beaker.

b. Stir the sample with a rubber policeman-tipped glass rod until all lumps are dissolved and any suspended insoluble matter is finely dispersed, heating gently if necessary.

c. Insert a rubber crucible holder into a 500 milliliter vacuum flask. Insert a dry, medium porosity sintered glass funnel previously weighed to the nearest 0.1 milligram.

d. Filter the sample through the sintered glass funnel using a vacuum. Use 100 milliliters of chlorobenzene to wash the residue from the beaker and the inside walls of the glass funnel. After releasing the vacuum, wash down the lower portion of the funnel, the crucible holder, and other areas as necessary with a few milliliters of chlorobenzene to remove all of the filtrate and collect in the vacuum flask.

e. Dry the funnel and contents 30 minutes at 149°C (300°F). Cool in a desiccator to room temperature. Weigh to the nearest 0.1 milligram.

f. Calculate percent filler as follows:

$$\% \text{ Filler} = \frac{(W_t - F - C)\ 100}{W}$$

W_t = weight of funnel plus filler plus celite

F = weight of funnel

C = weight of celite

W = weight of sample

4.6.4 Amine nitrogen. Amine nitrogen shall be determined by nonaqueous titration as specified below. Apparatus and reagent purity shall be per ASTM D 2073-62T, unless otherwise specified.

4.6.4.1 Preparation of 0.1 N perchloric acid solution (nonaqueous). Prepare a nonaqueous solution of 0.1 N perchloric acid by dissolving 8.5 milliliters of 70 percent perchloric acid in 1000 milliliters of glacial acetic acid. Then cautiously add 20 milliliters of acetic anhydride in small portions, mixing well after each portion.

WARNING

Perchloric acid in glacial acetic acid is strongly corrosive.

Concentrated perchloric acid is a powerful and unpredictable oxidizing agent. An explosion may result if it is allowed to contact organic materials such as plastics, wood, paper, cloth, and some chemicals.

4.6.4.2 Preparation of nonaqueous methyl violet indicator solution. Dissolve 0.1 gram of methyl violet in 100 milliliters of glacial acetic acid.

4.6.4.3 Standardization of perchloric acid solution. Standardize with duplicate samples of dried potassium acid phthalate as follows:

a. Weigh a 0.45 - 0.50 gram sample of dried potassium acid phthalate to the nearest 0.1 milligram into a 250 milliliter Erlenmeyer flask.
b. Add 50 milliliters of glacial acetic acid.
c. Dissolve the potassium acid phthalate solution by gentle heating.
d. Cool solution to room temperature and add 2 drops of methyl violet indicator solution.
e. Titrate with perchloric acid solution to a blue-green end point.
f. Make a blank determination on the reagents following the same procedure but omitting the potassium acid phthalate sample.
g. Calculate the concentration of the perchloric acid solution as follows:

$$N = \frac{W \times 1000}{(A-B)\ 204.2}$$

N = normality of the perchloric acid solution
W = weight of sample in grams
A = milliliters of perchloric acid solution required to titrate the sample
B = milliliters of perchloric acid solution required to titrate the blank.

4.6.4.4 Titration of curing agent. Titrate the curing agent with perchloric acid solution as follows:

a. Weigh a 0.20 - 0.30 gram sample of the curing agent to the nearest 0.1 milligram into a 250 milliliter beaker or Erlenmeyer flask. Add 50 milliliters of glacial acetic acid. Stir solution slowly with a magnetic stirrer.

b. Titrate with perchloric acid solution with constant stirring, using either methyl violet indicator solution or a potentiometric end point:
Methyl violet indicator: Add 2 drops of methyl violet indicator solution. Titrate with perchloric acid solution to a blue-green end point.
Potentiometric titration: Use a pH meter with glass and calomel electrodes. Plot incremental milliliters of perchloric acid solution versus the emf of the electrode system to obtain the equivalance point.

c. Make a blank determination on the reagents following the same procedure but omitting the curing agent sample.

d. Calculate the nitrogen value as follows:

$$\text{Percent amine nitrogen} = \frac{14N\ (A-B)}{10\ W}$$

4.6.5 Viscosity. Viscosity shall be determined per ASTM D1084-55T Method B, using a No. 7 spindle at 20 rpm for the adhesive base and a No. 1 spindle at 20 rpm for the curing agent.

4.6.6 Infrared spectrum.

4.6.6.1 Sample preparation. Place 5 - 10 grams of the adhesive base in a beaker of suitable size and cover with a reagent grade acetone. Mix the adhesive base with the acetone until the particles of filler are free from resin. Decant or centrifuge the acetone portion containing the resin. Remove most of the acetone by gentle heating on a steam bath. Place a drop of the remaining material on an NaCl plate and warm gently with an infrared lamp to effect complete removal of the acetone solvent. Place a second NaCl plate over the specimen and apply gentle pressure to obtain a clear, uniform film of resin between the plates.

4.6.6.2 Testing. Take an infrared spectrum of the specimen from 1 - 15 microns wavelength using an infrared spectrophotometer. A Perkin-Elmer No. 221 has been found to be satisfactory.

4.6.6.3 Analysis. The infrared spectrum of the specimen shall be compared to the spectrum shown in Figure 1 except that any absorption at 5.85 microns due to residual acetone solvent shall be ignored. Any spurious absorption peaks in excess of an absorbance of 0.05 shall be cause for rejection.

4.6.7 Vacuum weight loss. Vacuum weight loss shall be determined per JPL Specification ZTF-4010-0007 using the following test parameters:

a. Temperature. 150 ±2°C (302 ±3°F).
b. Pressure. 10^{-5} torr maximum.
c. Specimens. The adhesive shall be cured in place on Type 2024-T3 aluminum sheet of suitable thickness. The thickness and weight of the cured adhesive shall be 0.015-0.020 inch and 1.0-2.0 gram, respectively. The aluminum sheet shall be prepared per JPL Specification GMO-50234-PRS before applying the adhesive.

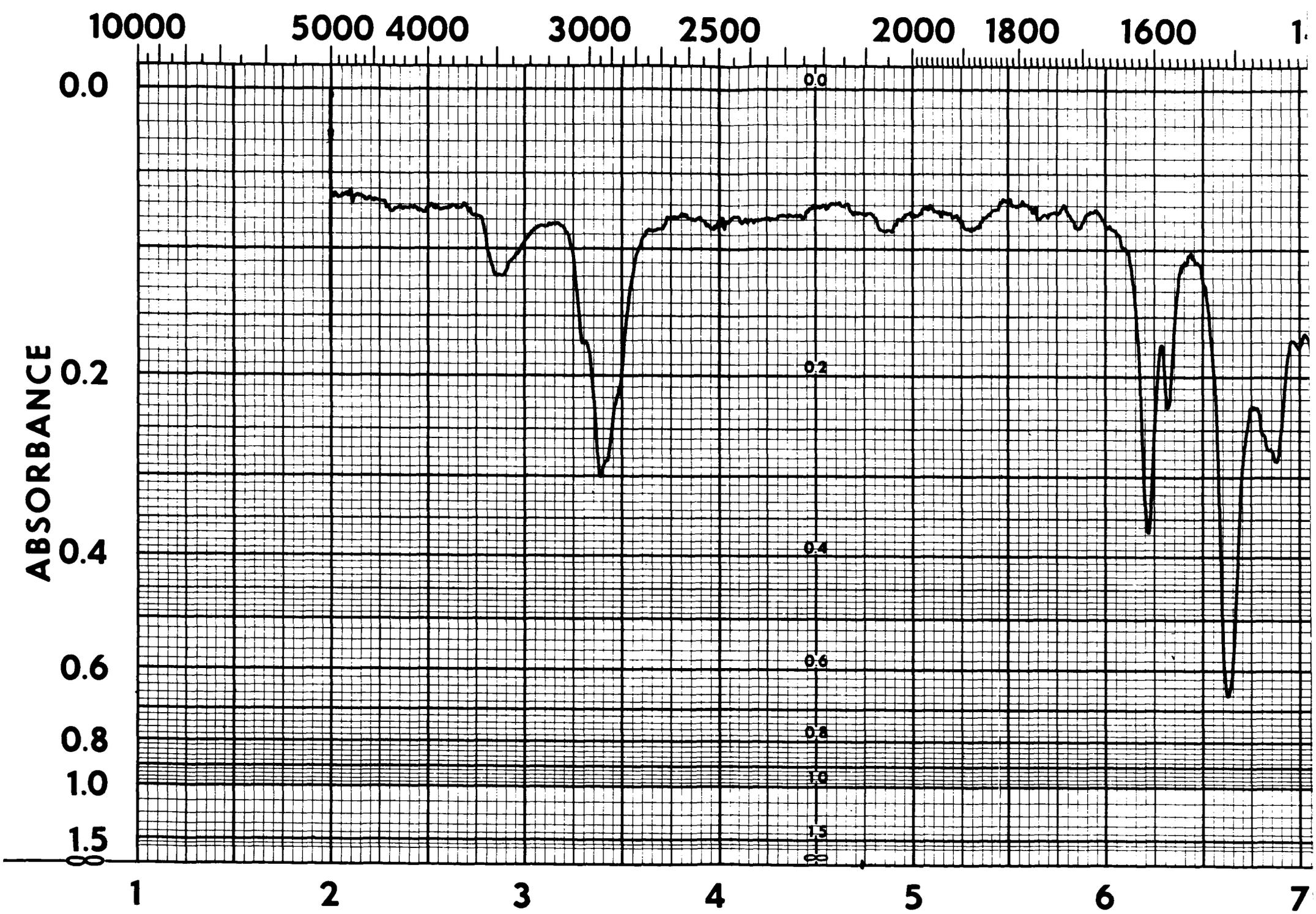
4.6.8 Shelf life. A sufficient quantity of adhesive base and curing agent to perform the tests required in this paragraph shall be stored at a minimum temperature of 25°C (77°F) and a minimum time of 6 months. At the end of this time, the adhesive base and curing agent shall be tested for conformance to the viscosity requirements of Table II and the numbers 1 and 2 tensile shear requirements of Table III.

5. PREPARATION FOR DELIVERY

Not applicable.

6. NOTES

6.1 Intended use. Adhesives conforming to this specification are intended for use in bonding spacecraft structural components. The specification is written to control the quality and consistency of the adhesive from batch to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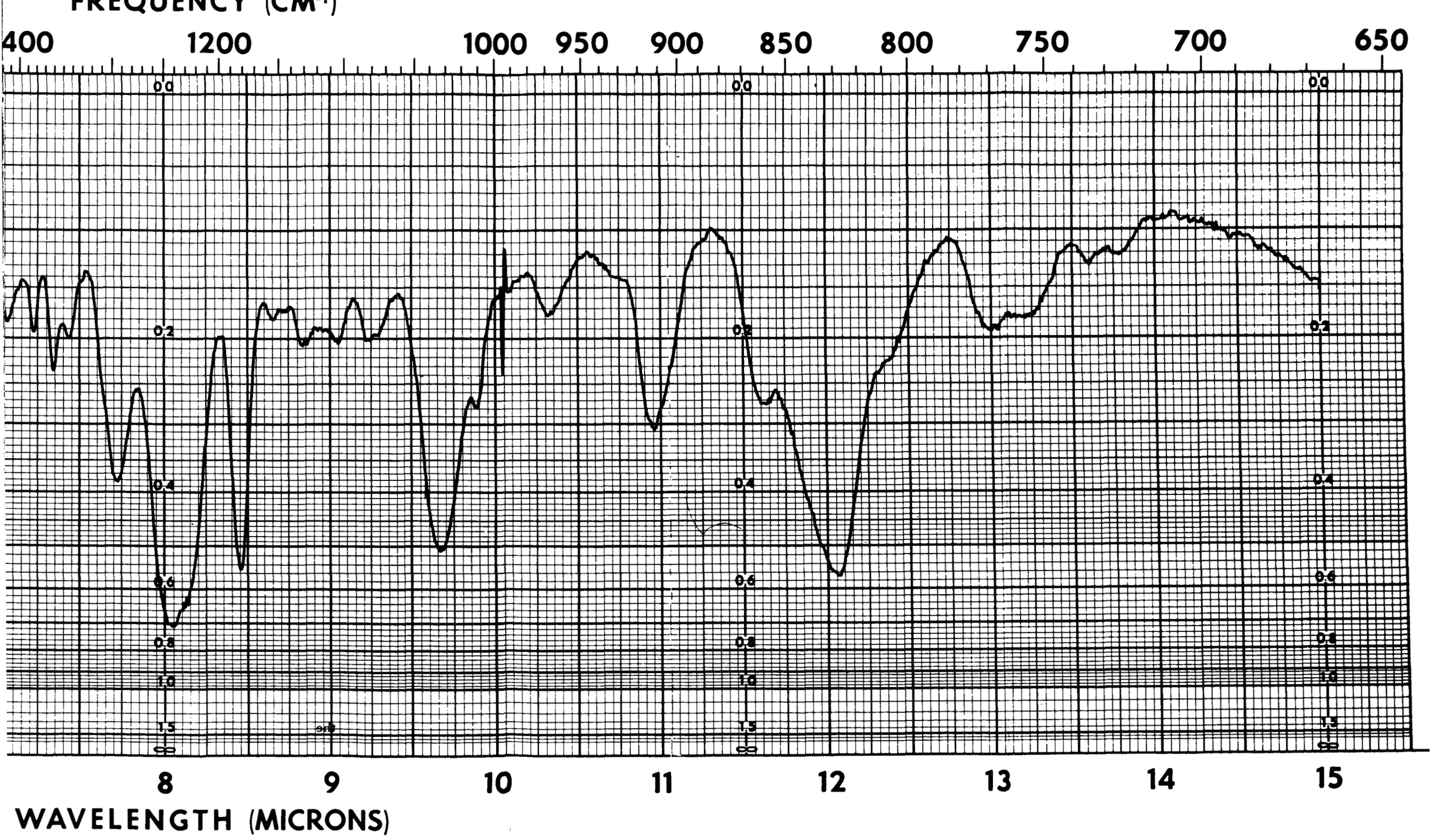

Figure 1. Infrared Spectrum

batch rather than to define its use in bonding a particular adherend combination. In general, a wide variety of materials may be bonded. The use of the adhesive in bonding any adherend or any adherend combination should be substantiated by test and a process specification released to cover its use in each application. The following factors should be taken into account when using the adhesive:

a. Each adherend material type may require substantially different surface preparation methods to effect satisfactory bond strength. Surface preparation methods may vary widely from a simple solvent wipe to a complicated multiple chemical etch. Surface abrasion methods may include hand sanding, sand blasting, and vapor honing, each with a wide variety of grit types and sizes. No one method or combination of methods will provide a universal surface preparation system.

b. Bonding of dissimilar materials may yield low bond strengths because of difference in coefficient of thermal expansion.

c. The adhesive may be incompatible with an adherend resulting in poor wetting or an undesirable chemical reaction.

d. The viscosity and/or the curing method of the adhesive may be undesirable or incompatible for a particular bonding operation.

6.2 Definitions.

a. Batch. That quantity of material which has been manufactured at one time or subjected to some unit chemical or physical mixing process intended to make the final product homogeneous.

b. Adhesive base. The component of the two component adhesive system that consists of an epoxy resin and filler.

c. Curing agent. The component of the two component adhesive system that consists of one or more aromatic amines.

d. Mixed adhesive. The mixture of adhesive base and curing agent specified in the instruction sheet.

e. Cured adhesive. Mixed adhesive that has been exposed to the curing time and temperature specified in the instruction sheet (3. 5. 3).

f. Adhesive and adhesive materials. Refers collectively to adhesive base, curing agent, mixed adhesive, and/or cured adhesive.

g. Pot life. The time after mixing together the adhesive base and the curing agent during which the mixed adhesive still meets the application requirements and the adhesive cured therefrom meets all of the applicable requirements of this specification.

6. 3 Safety. The adhesive base contains epoxy resin which may produce contact dermatitis following frequent or prolonged exposure with the skin. The amine curing agent may also produce sensitization dermatitis and, in addition, prolonged contact may produce caustic burns. The vapors of both materials are irritating to the eyes and mucous membranes of the respiratory tract. The following precautions should properly be observed:

a. Mix in an area with adequate ventilation to remove fumes.

b. Wear rubber gloves when necessary to prevent skin contact.

c. Wear goggles or shields when necessary to provide eye protection.

d. Cure in ovens vented to the outside atmosphere or vented to a room with adequate ventilation.

e. In case of accidental contact with adhesive or curing agent, wash contaminated skin areas immediately with soap and water. (Use of solvents to cleanse contaminated skin areas should be kept to an absolute minimum.)

f. Launder contaminated clothing before reuse.

g. Prevent contamination of areas such as door knobs, valves, handrails, etc.

6.4 Qualified products. The following product has been qualified to the requirements of this specification and is approved for purchase subject to acceptance of the specification by the supplier:

Product	Manufacturer
Epon Adhesive 901 with curing agent B-3	Shell Chemical Co.